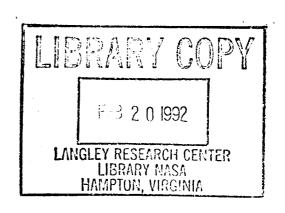
NASA Technical Memorandum 104743

NASA-TM-104743 19920008158

# Long-Term Corrosion Evaluation of Stainless Steels in Space Shuttle Iodinated Resin and Water

Douglas D. Krohn

January 1992





 	_	-	-	 	
					-
					-
					·
					•
					¥
					•
 <del></del>				 <del></del>	

## NASA Technical Memorandum 104743

Long-Term Corrosion Evaluation of Stainless Steels in Space Shuttle Iodinated Resin and Water

Douglas D. Krohn Lyndon B. Johnson Space Center Houston, Texas

National Aeronautics and Space Administration Lyndon B. Johnson Space Center Houston, Texas

January 1992

			-
			•
			-

# **CONTENTS**

Section		Page
1.0 2.0	ABSTRACT INTRODUCTION	1 1
3.0 3.1 3.2	TEST ARTICLES STAINLESS STEEL TUBES IN AQUEOUS SOLUTION STAINLESS STEEL STRIP IN MOIST IODINE RESIN	2 2 2
4.0 4.1 4.2	TEST CONDITIONS STAINLESS STEEL TUBES IN AQUEOUS SOLUTION STAINLESS STEEL STRIP IN MOIST IODINE RESIN	2 2 4
5.0 5.1 5.1.1 5.1.2	ANALYSIS CHEMICAL ANALYSIS OF AQUEOUS SOLUTION Procedure Results	5 5 5 5
5.2 5.2.1 5.2.1.1 5.2.1.2 5.2.2 5.2.2.1 5.2.2.1.1 5.2.2.1.2	METALLURGICAL ANALYSIS OF SPECIMENS Procedures Stainless Steel Tubes In Aqueous Solution Stainless Steel Strip In Moist Iodine Resin Results Stainless Steel Tubes In Aqueous Solution SEM analysis Metallography analysis	88889999
5.2.2.2 5.2.2.2.1 5.2.2.2.2 5.2.2.2.3	Stainless Steel Strip In Moist Iodine Resin SEM analysis Metallography analysis EDS analysis	18 18 18 18
6.0	DISCUSSION AND CONCLUSIONS	21
7.0	REFERENCES	22

# **TABLES**

Table		Page
1.	Test Conditions of Stainless Steel Specimens Exposed to	
	Aqueous lodine	3
2.	Moist MCV Resin Specimen Conditions	3
3.	Chemical Analysis Results	6
4.	EDS Analysis of Stainless Steel Strip Exposed to Moist MCV	
	Resin	18
	<u>FIGURES</u>	
Figure		Page
1.	Stainless steel specimen in moist MCV resin.	4
2.	Aqueous iodine exposure - SS 316L O.D. SEM at 500x.	10
3.	Aqueous iodine exposure - SS 316L I.D. SEM at 500x.	10
4.	Aqueous iodine exposure - SS 321 O.D. SEM at 500x.	11
5.	Reference specimen - SS 321 O.D. SEM at 500x.	11
6.	Aqueous iodine exposure - SS 321 I.D. SEM at 500x.	12
7.	Reference specimen - SS 321 I.D. SEM at 500x.	12
8.	Aqueous iodine exposure - SS 347 O.D. SEM at 500x.	13
9.	Reference specimen - SS 347 O.D. SEM at 500x.	13
10.	Aqueous iodine exposure - SS 347 I.D. SEM at 500x.	14
11.	Reference Specimen - SS 347 I.D. SEM at 500x.	14
12.	Aqueous iodine exposure - SS 316L sectioned metallograph at 400x.	15
13.	Aqueous iodine exposure - SS 321 sectioned metallograph at 400x.	16
14.	Reference specimen - SS 321 sectioned metallograph at 400x.	16
15.	Aqueous iodine exposure - SS 347 sectioned metallograph at 400x.	17
16.	Reference specimen - SS 347 sectioned metallograph at 400x.	17
17.	Moist MCV resin exposure - stainless steel (301) SEM at 500x.	19
18.	Moist MCV resin exposure - stainless steel (301) SEM at 500x.	19
19.	Moist MCV resin exposure - stainless steel (301) sectioned metallograph at 400x.	20

# **ACRONYMS**

EDS Energy Dispersive Spectroscopy

IWS Integrated Water System

JSC Johnson Space Center

JEOL Japan Electronic Optic Laboratory

LCV Leuco Crystal Violet

MCV Microbial Check Valve

meq Milliequivalents

mpy Mils per year

R2A Growth medium used in performing heterotrophic bacteria counts

SEM Scanning Electron Microscopy

USP United States Pharmacopoeia

		-
		•

## 1.0 ABSTRACT

The effects of stainless steel exposure to iodinated water is a concern in developing the Integrated Water System (IWS) for Space Station Freedom. The IWS has a life requirement of 30 years. However, the effects of general and localized corrosion over this length of time have not been determined for the candidate materials of interest. In 1978, Umpqua Research Center immersed stainless steel 316L, 321, and 347 specimens in a solution of deionized water and Space Shuttle Microbial Check Valve Resin. A decision was made in April of 1990, to analyze the effects of this exposure. Chemical analysis of the solution was performed to determine the level of corrosion formed over this time period. In addition, the surface of each specimen was examined with scanning electron microscopy and metallography to determine the extent of general and pitting corrosion. This study showed that the attack on the stainless steels was negligible and never penetrated past the first grain boundary layer. Of the three alloys evaluated, 316L performed the best, however, all three materials proved to be compatible with an aqueous iodine environment.

In addition to the specimens exposed to aqueous iodine, a stainless steel specimen, (unspecified alloy) was exposed to moist microbial check valve resin and air. This environment allowed contact of the metal to the resin as well as to the iodine vapor. The exposure period of this metal was thought to be 11 years as with the other specimens. Since the particular stainless steel alloy was not known, energy dispersive spectroscopy was used to determine that this alloy was stainless steel 301. Intergranular corrosion was found on the specimen; however, the corrosion was limited to the first grain boundary layer.

## 2.0 INTRODUCTION

Umpqua Research Company, of Myrtle Creek, Oregon, immersed 316L, 321, and 347 stainless steel coupons in Microbial Check Valve (MCV) resin and water in September of 1978. These coupons had been exposed to this environment for approximately 11 years and 6 months before they were removed for analysis. This study was conducted to investigate the corrosive effect iodinated water and MCV resin had on these various stainless steel alloys. The goal was to provide better insight into the criteria required for the selection of materials in the Space Station Freedom IWS. The specific objectives of the study were to determine the:

- 1. Aqueous metal ion concentrations in each solution
- 2. Aqueous cation and anion constituents in each solution
- 3. Corrosion rates of each individual metal specimen
- 4. Extent of pitting corrosion on the metal surface

Umpqua Research Company performed an analysis of the chemical constituents present in each solution. This included analysis of iodine, metal ion, anion, and cation concentration levels. Following Umpqua's work, the metal coupons were sent to the

Johnson Space Center (JSC) for surface analysis. The surface analysis evaluated the degree of both general and pitting corrosion present on each specimen.

In the course of initiating this study, a vial was found that contained a strip of an undetermined stainless steel alloy. This alloy was in contact with the moist MCV resin and the air trapped in the vial. It was decided that this specimen would be analyzed in conjunction with the other specimens to determine the effects that iodine vapor and MCV resin had on stainless steel. Surface analysis was used to evaluate the extent of corrosion as well as to determine the particular stainless steel alloy.

## 3.0 TEST ARTICLES

## 3.1 STAINLESS STEEL TUBES IN AQUEOUS SOLUTION

The test articles used in the aqueous exposure study were prepared from 1/4" stainless steel tubing. Stainless steel 316L, 321, and 347 were all sectioned in 2-4 cm lengths. Table 1 presents dimension, weight, and volume data which was obtained after the exposure period. Due to the low level of corrosive attack which was observed in the analysis, the specimen dimensions are not expected to have changed over the test period. An accurate measurement of the length could not be made due to the uneven ends of each specimen. As a result, the equivalent length of each sample was determined from the calculated cross sectional area and the measured weight and density of the specimen. Surface area was based on the diameter and length dimensions and did not take into account the cross-sectional area of both ends of the tube.

Samples of 321 and 347 cut from the same pieces of tubing as the test coupons but not exposed to the iodine environment were available and analyzed for comparative purposes. A reference sample of 316L from the original stock was not available.

#### 3.2 STAINLESS STEEL STRIP IN MOIST IODINE RESIN

The test article used in the moist iodine exposure study consisted of a strip of unknown stainless steel in contact with moist MCV resin and air. Table 2 lists the weight and dimensions of the specimen. The dimensions are approximate since both the length and width of the sample were variable. The surface area was calculated from sample weight and thickness measurements.

# 4.0 TEST CONDITIONS

#### 4.1 STAINLESS STEEL TUBES IN AQUEOUS SOLUTION

Three Pyrex disposable culture vials, 16 x 125 mm with rubber lined plastic caps, were used to hold the aqueous solutions and test articles in a sealed environment.

TABLE 1. TEST CONDITIONS OF STAINLESS STEEL SPECIMENS EXPOSED TO AQUEOUS IODINE

Parameter	Blank	316L	321	347
Specimen weight (g)	-	1.7133	2.9934	2.3155
Specimen dimensions ( Outside diameter Inside diameter Equivalent length <sup>a</sup>	cm) - - -	0.6350 0.5144 1.96	0.6350 0.5144 3.43	0.6350 0.5144 2.66
Surface area (cm²)b	-	7.08	12.37	9.60
Wet resin weight (g)	6.73	3.84	5.37	5.22
Liquid volume (ml)	2.54	3.55	4.30	5.54

<sup>&</sup>lt;sup>a</sup>Equivalent length based on specimen density, weight, inner diameter, and outer diameter.

TABLE 2. MOIST MCV RESIN SPECIMEN CONDITIONS

Parameter	Value	
Specimen weight (g) Specimen dimensions (cm)	1.947	
Width	0.508 - 0.610	
Approximate length	7.366	
Thickness	0.0648	
Surface area (cm²)a	7.490	

<sup>&</sup>lt;sup>a</sup>Surface area based on specimen density, weight, and thickness.

bSurface area based on equivalent length, inner diameter, and outer diameter.

A blank vial was prepared to determine the effects of the Pyrex glass and MCV resin on the chemical constituents in the solution. The water was deionized and had a conductivity of less than 1 μohm/cm. Oxygen was not removed from the water; therefore, approximately 9 parts per million (ppm) oxygen was initially present in each vial. The MCV resin (Batch #80511) was prepared according to Umpqua Standard Procedures SP 102 (Appendix), which meets the Umpqua Iodinated Resin Specification CPS-100. Food-grade quality raw resin was used to manufacture the MCV resin. The iodine and iodide reagents used were USP grade. Although initial iodine and iodide concentration levels were not measured, the iodinated resin used is well characterized. It is expected that the initial iodine concentration was 2-4 mg/l and the iodide concentration was approximately 0.2 mg/l. Each vial contained an unmeasured quantity of trapped air above the solution. The vials were contained in a test tube rack located on various open shelves in Umpqua's chemistry laboratory. These vials had not been opened since they were originally prepared in September of 1978.

#### 4.2 STAINLESS STEEL STRIP IN MOIST IODINE RESIN

Included with the specimens exposed to iodine in aqueous solution was a vial that contained a strip of unknown stainless steel in contact with moist MCV resin and air. This specimen was exposed to this environment for the same period of time as the tube specimens. Since free water was not present, a chemical analysis was not performed. Figure 1 shows the specimen configuration in the Pyrex vial.

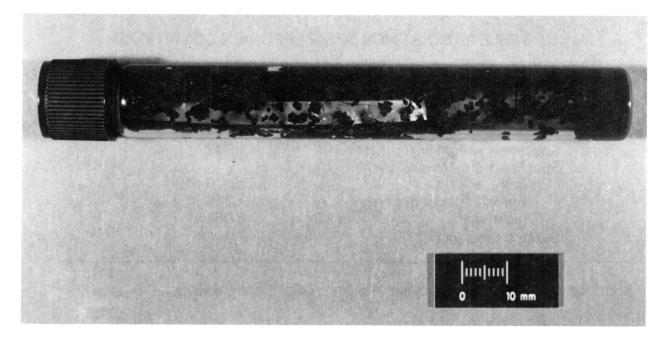


Figure 1. Stainless steel specimen in moist MCV resin.

## 5.0 ANALYSIS

## 5.1 CHEMICAL ANALYSIS OF AQUEOUS SOLUTION

## 5.1.1 Procedure

The chemical analysis was performed by Umpqua Research Company. Iodine and iodide analysis were performed per SM<sup>1</sup> 4500 I-B. Metal ion analysis for manganese was conducted following EPA<sup>2</sup> 243.2. Metal ion analysis for nickel, molybdenum, and chromium was performed per SM<sup>1</sup> 3113. Iron analysis was conducted utilizing EPA<sup>2</sup> 236.2. Cation analysis of potassium, sodium, calcium, and magnesium was performed following SM<sup>1</sup> 3111. Heterotrophic plate counts were performed for the microbial analysis by incubating water samples on R2A auger (growth medium) for 7 days at 20°C. These steps were followed in performing the chemical analysis:

- 1. Weigh each vial.
- 2. Mark liquid level on each vial.
- 3. Make a solution of iodine that has the same color as samples and determine approximate concentration.
- 4. Remove 0.5 ml from each vial with a sterile pipet and spread on R2A plate.
- 5. Incubate R2A plates at 20°C for 7 days.
- 6. Take 1.0 ml from each vial and dilute to 10.0 ml.
- 7. Remove 1.0 ml from each 1/10 dilution and dilute to 10.0 ml.
- 8. Test diluted samples for Fe, Cr, Ni, Mn, Mo. If below detection levels, test undiluted sample.
- 9. To determine iodine levels, set up a Leuco Crystal Violet (LCV) method that uses 3 ml samples.
- 10. Measure iodide and iodine on appropriate dilutions.
- 11. Remove metal samples, rinse in deionized water and blot dry.
- 12. Weigh each sample.
- 13. Place each sample in plastic bag purged with nitrogen and seal.
- 14. Drain liquid from each tube into graduated cylinder and record volume.
- 15. Measure pH and conductivity.
- 16. Take sample and perform cation analysis for each solution.
- 17. Weigh vial containing resin.
- 18. Use solution to rinse resin from each vial and weigh empty vial.
- 19. Return resin and solution to each vial and seal.
- 20. Send metal samples to NASA/JSC for surface tests.

#### 5.1.2 Results

The results of the chemical analysis performed by Umpqua Research Company are presented in Table 3. The first finding of interest was that the iodine concentration level ranged from 70 to 80 mg/l in the test vials but was 236 mg/l in the blank. These high readings were unexpected since it was thought that the MCV resin would maintain an iodine concentration around 10 mg/l. The iodide level was also very high

TABLE 3. CHEMICAL ANALYSIS RESULTS

	_				Vial			
Parameter	1	Blank		316L		321	<u>.                                    </u>	347
рН		3.19		3.20		3.26		3.33
Specific conductivity (µmho/cm)	1	3000	ı	9000	;	9100	• ·	7400
Microbial analysis	<2 CF	-U/ml	<2 CF	U/ml	<2 CF	U/ml	<2 CF	U/ml
lodine (mg/l)		236		81.1		80.6		69.6
Metals (mg/l):								
Mn	(	0.103		1.57	C	0.360		2.79
Ni		0.04		0.48		0.88		0.57
Fe		1.22		2.34		4.74		2.28
Мо	ND@	0.002	(	0.047	ND@0	0.002	C	.015
Cr	(	0.156	(	).316	(	).976	C	.612
Anions (mg/l)/(meq)	:							
ŀ	11400/	89.8	6630/	52.2	6510/	51.3	4530/	35.7
Total anions (meq)		89.8		52.2		51.3		35.7
Cations (mg/l)/(med	ı):							
K+	1290/	33.0	646/	16.5	762/	19.5	547/	14.0
Na+	380/	16.5	17/	0.73	10/	0.43	17/	0.74
Ca++	414/	20.6	382/	19.1	425/	21.2	342/	17.1
Mg++	16/	1.3	16/	1.3	10/	0.78	11.3/	0.93
H+	3.1/	3.1	2.5/	2.5	2.6/	2.6	2.4/	2.4
Total cations (meq)		74.3		40.1		44.5		35.2

(over 11,000 mg/l in the blank and between 4500 and 6500 mg/l in the test vials). Since the original iodide concentration was less than 1 mg/l, these high iodide concentrations indicate that a significant conversion of iodine (l<sub>2</sub>) to iodide (l<sup>-</sup>) occurred over the 11-year time period. The cause of this conversion process is unknown; however, interactions of iodine with the metal specimens, glass vials, MCV resin, and rubber lining in the plastic caps may have been a contributing factor. The increased amount of MCV resin in the blank vial may have resulted in iodine and iodide concentrations that were over twice that of the test vials. The ratio of wet resin weight to liquid volume in the test vials ranged from 0.94 to 1.25 which is less than half that of the blank (2.65 in the blank).

As a result of the unexpectedly high iodide concentrations, a cation analysis was made to determine what chemical species were present in solution to balance the anion charge resulting from the iodide. Sodium, calcium, magnesium, and potassium were all found in significant amounts. The potassium concentration demonstrated the same phenomenon between the test vials and the blank as both iodine and iodide concentrations. The most probable source of potassium is from the MCV resin since potassium iodide is used to prepare the resin. Since the ratio of resin to liquid volume in the blank was over twice that of the test vials, it is reasonable that the potassium concentration in the blank was approximately twice that of the test vials. The levels of calcium and magnesium were similar for all vials and may have originated from the glass walls. The sodium concentration of the blank was over 20 times higher than in the test vials. This high sodium concentration in the blank has not been explained, but the possibility of the blank being contaminated prior to the chemical analysis should be considered. The milliequivalents<sup>1</sup> (meg) for both the anion (iodide) and cation constituents found in this analysis show that a majority of the ions in solution have been accounted for. The measured cation charge is approximately 80% of the anion charge in the blank and 316L vial, 87% in the 321 vial, and 99% in the 347 vial.

Significant amounts of iron, nickel, manganese, and chromium were found in the blank vial. These contaminants may have originated from either the glass or MCV resin. Even if the blank values are not factored into the results in each test vial, the level of metal corrosion is low. Corrosion rates for all three specimens were calculated without correcting for the blank values. This was performed by converting the metal concentrations into the equivalent volume of metal lost from each specimen. The specimen surface area and the time exposure were then used to obtain a corrosion rate. This corrosion rate was on the order of 1 x 10<sup>-5</sup> mils per year (mpy) for all three specimens. This level of general corrosion is negligible, although it does not eliminate the possibility that localized corrosion such as pitting may still be present. The highest metal concentration was 4.74 mg/l of iron in the stainless steel 321 vial. The 316L vial had the lowest concentration of metals: 2.34 mg/l iron and 1.57 mg/l manganese made up the majority of metals in this solution. The predominant species in the 347 vial consisted of 2.79 mg/l manganese and 2.28 mg/l iron. High levels of manganese were found in all the test vials. Manganese concentrations in these alloys are typically less than 2%. The explanation for manganese preferentially going into solution is not known.

<sup>1</sup> Milliequivalent = Ion concentration (mg/l) x Ion charge / Ion molecular wt.

## 5.2 METALLURGICAL ANALYSIS OF SPECIMENS

#### 5.2.1 Procedures

Surface analysis of each coupon was performed at the JSC Materials Branch. Scanning Electron Microscopy (SEM) analysis was performed on a JEOL JSM 820 and an AMRAY model 1400. Metallography on each specimen was performed on a Bausch and Lomb Research II Metallograph.

#### 5.2.1.1 Stainless Steel Tubes In Aqueous Solution

This analysis was performed with the intent of evaluating the susceptibility of each alloy to both general and pitting corrosion. The specimens were examined as received from Umpqua in sealed plastic bags. The only surface cleaning performed was a rinsing of the specimens in deionized water. The outer surface of each specimen was first examined under a stereoscope to determine particular areas of interest. The specimen was then transferred to the SEM and the outer surface was examined. To investigate the inner surface, a 1/4 inch end was cut from each specimen and sectioned in half down its axis. Following SEM analysis, a specimen cross-section was mounted and polished for metallography analysis. This was performed to determine the extent of pitting on each specimen. The detailed procedures were as follows:

- 1. Stereoscopic scan of specimen, determine areas of interest.
- 2. SEM scan tube exterior and interior where possible.
- 3. Photograph exterior surface at 12, 100, 200, and 500 magnification.
- 4. Cut 1/4" section of tube at area of interest.
- 5. Section 1/4" specimen in two (half circles).
- 6. SEM scan and photograph inner surface at 100, 200, and 500 magnification.
- 7. Mount the sectioned specimen, and perform metallography to investigate pitting.
- 8. Photograph at 400 magnification.

#### 5.2.1.2 Stainless Steel Strip In Moist Iodine Resin

The procedure followed was similar to that above. Unlike the others, this specimen was shipped to JSC in the test vial. The only surface cleaning involved rinsing the specimen with deionized water to remove the MCV resin and the iodine from the surface. Both sides of the specimen were exposed to the same environment, therefore, neither side was differentiated from the other. Since the type of alloy was unknown, the actual composition was determined using an energy dispersive spectroscopy (EDS) analysis technique. The step-by-step procedure was as follows:

- 1. Remove specimen from vial.
- 2. Rinse specimen with deionized water and dry.
- 3. Obtain weight and dimensions.
- 4. Scan specimen with stereoscope and determine areas of interest.
- 5. SEM scan and photograph specimen.
- 6. Mount a section and perform metallography to evaluate pitting.
- 7. EDS the sectioned specimen to determine type of stainless steel.

#### 5.2.2 Results

#### 5.2.2.1 Stainless Steel Tubes In Aqueous Solution

5.2.2.1.1 <u>SEM analysis</u>. Figures 2 and 3 show the outer and inner surfaces, respectively, of the 316L specimen. The outer surface has a rougher surface finish than the inner surface. This was apparent on all three specimens examined and is probably a result of the tube extrusion process. Since a reference specimen from the original tube stock was not found for the 316L, a comparison could not be made between its finish and that of the original material. The outer surface had scratches that ran longitudinally with the tube axis. These markings were probably due to the drawing process used to manufacture the tube. The presence of these markings indicate that the level of corrosion has been small enough not to change the original surface finish.

Figures 4 through 7 show a comparison of the 321 exposed specimen surfaces to the reference specimen. The outer surface of both the test and reference specimens are similar in nature. Both have scratches that run longitudinally with the tube axis. As with the 316L, the origin of these marks probably resulted from the tube drawing process. The level of corrosion due to its exposure in iodinated water was relatively small since these markings are clearly present on the surface of the test specimen.

Both the test and reference 321 specimens featured inclusions on their inner surfaces. Figure 6 (test specimen) shows a long inclusion that runs the vertical length of the photograph. This particular inclusion is relatively free of the stringer material that created the inclusion. Figure 7 (reference specimen) also has an inclusion at the top center region of the photograph. Part of this inclusion stringer material has remained. Evidence of corrosion around the various inclusions investigated was not found. In general, the types of inclusions found on both specimens were similar.

Figures 8 through 11 compare the outer and inner surface of the 347 specimen to that of its reference specimen. As with the other alloys, no apparent change is observed in the surface features between the exposed and reference specimens. Surface drawing marks are still present on the outer diameter of the exposed specimen. As with the other specimens, this indicates that the surface finish has not changed significantly.

5.2.2.1.2 <u>Metallography analysis</u>. Figures 12 through 16 are micrographs of metallography performed on each alloy. They show areas in which pits were found. The majority of the cross-sections were relatively free of pitting. No observable difference was found between the inner and outer surfaces. The pits shown do not penetrate past the first grain boundary layer. In addition, both test and reference specimens have similar surface roughness. This is an indication that a minimal level of corrosion has occurred. Furthermore, the grain structure does not indicate the presence of intergranular corrosion.



Fig. 2. Aqueous iodine exposure - SS 316L O.D. SEM at 500x.

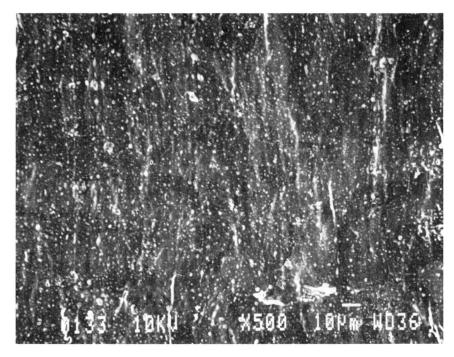


Fig. 3. Aqueous iodine exposure - SS 316L I.D. SEM at 500x.

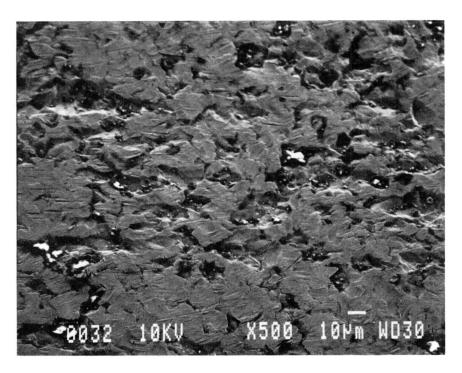


Fig. 4. Aqueous iodine exposure - SS 321 O.D. SEM at 500x.



Fig. 5. Reference specimen - SS 321 O.D. SEM at 500x.

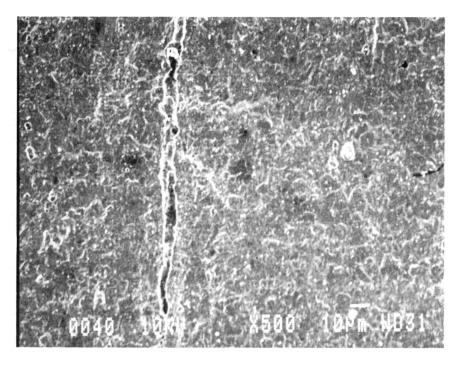


Fig. 6. Aqueous iodine exposure - SS 321 I.D. SEM at 500x.

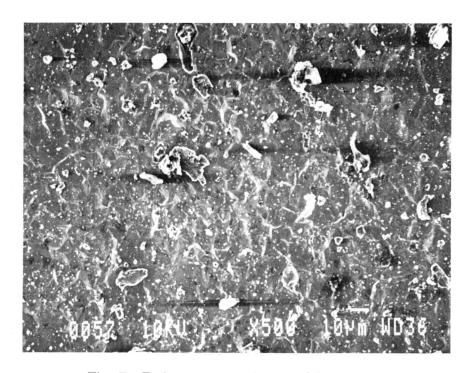


Fig. 7. Reference specimen - SS 321 I.D. SEM at 500x.

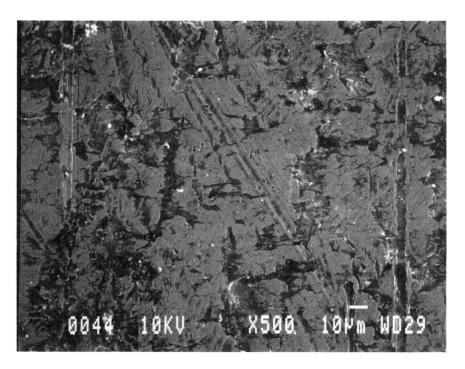


Fig. 8. Aqueous iodine exposure - SS 347 O.D. SEM at 500x.

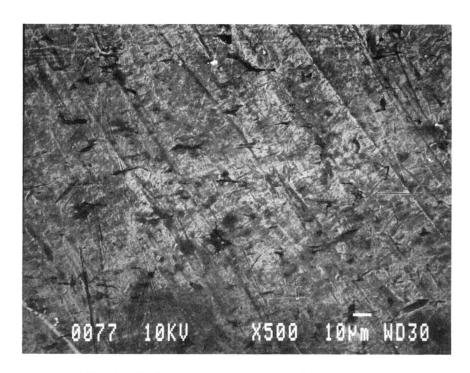


Fig. 9. Reference specimen - SS 347 O.D. SEM at 500x.

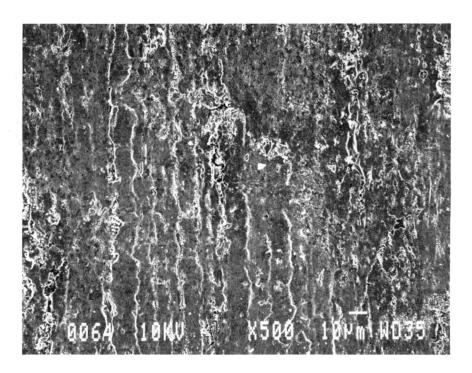


Fig. 10. Aqueous iodine exposure - SS 347 I.D. SEM at 500x.

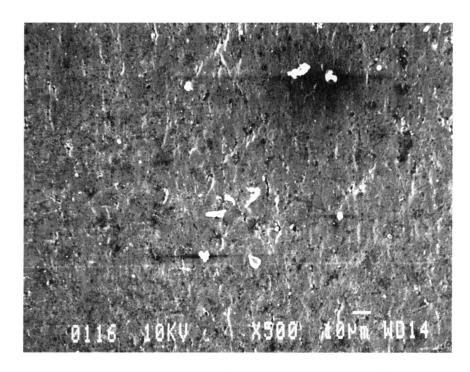


Fig. 11. Reference Specimen - SS 347 I.D. SEM at 500x.

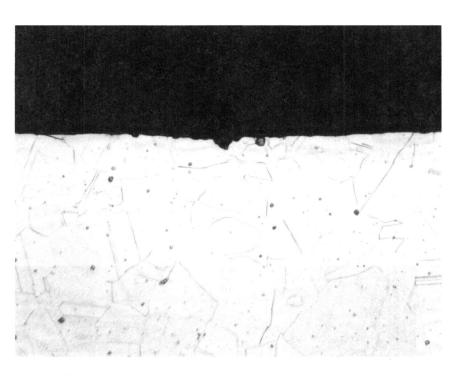


Fig. 12. Aqueous iodine exposure - SS 316L sectioned metallograph at 400x.



Fig. 13. Aqueous iodine exposure - SS 321 sectioned metallograph at 400x.

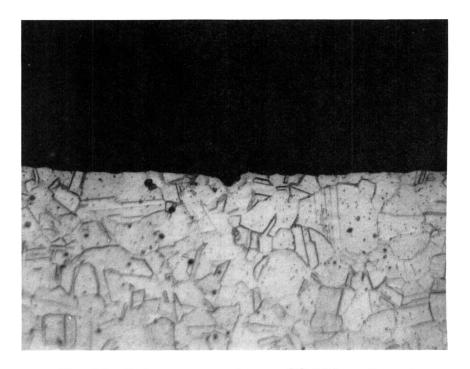


Fig. 14. Reference specimen - SS 321 sectioned metallograph at 400x.

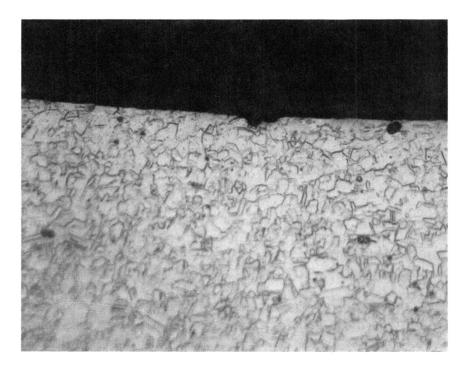


Fig. 15. Aqueous iodine exposure - SS 347 sectioned metallograph at 400x.

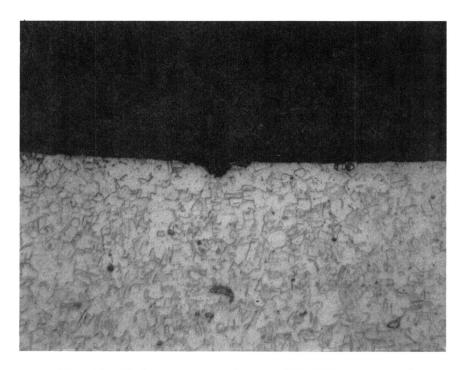


Fig. 16. Reference specimen - SS 347 sectioned metallograph at 400x.

#### 5.2.2.2 Stainless Steel Strip In Moist Iodine Resin

- 5.2.2.2.1 <u>SEM analysis</u>. Figures 17 and 18 show the surface features of the unmarked stainless steel specimen after it was rinsed off with deionized water. The dark areas were analyzed using EDS. These areas were high in potassium, iodine, chlorine, silicon, and molybdenum. These products probably resulted from the iodine resin and, to a lesser extent, corrosion products from the stainless steel. Figure 18 illustrates a ring of the dark material that was formed on the surface. This may have been where a resin pellet adhered to the surface of the specimen for an extended period of time. The ridges on the metal surface were not found on the specimens exposed to the aqueous environment. These ridges indicate that an intergranular attack is taking place at the grain boundaries of the metal.
- 5.2.2.2.2 <u>Metallography analysis</u>. Figure 19 is the sectioned metallograph micrograph of the specimen. The results of intergranular corrosion are evident at the grain boundaries on the surface of the metal. This attack is present in the first grain boundary but does not appear to progress further into the specimen.
- 5.2.2.3 <u>EDS analysis</u>. The stainless steel specimen used in the metallography was analyzed to determine the particular stainless steel alloy that was used. Table 4 lists the analyzed composition for the specimen. In comparing this analysis to the nominal compositions of various AISI grades of stainless steels, the closest match appeared to be SS 301.

TABLE 4. EDS ANALYSIS OF STAINLESS STEEL STRIP EXPOSED TO MOIST MCV RESIN

Element	Specimen wt%	SS 301 wt%	
Si	1.23	1.00	
Мо	0.81	-	
Cr	17.62	16-18	
Mn	0.94	2.00	
Fe	72.93	71-75	
Ni	6.47	6-8	

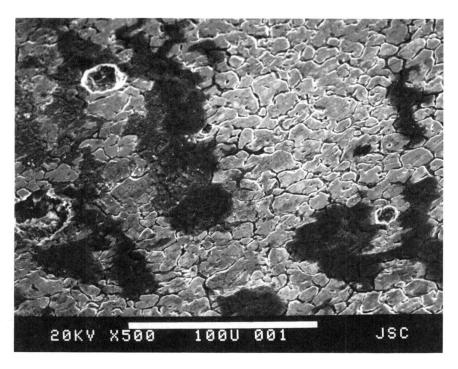


Fig. 17. Moist MCV resin exposure - stainless steel (301) SEM at 500x.

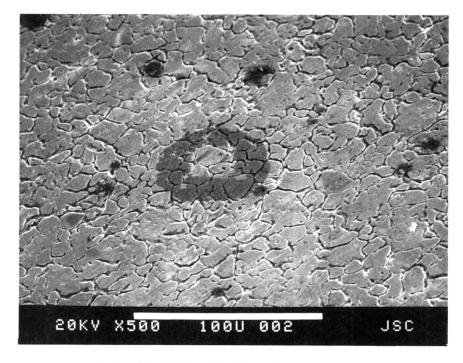


Fig. 18. Moist MCV resin exposure - stainless steel (301) SEM at 500x.

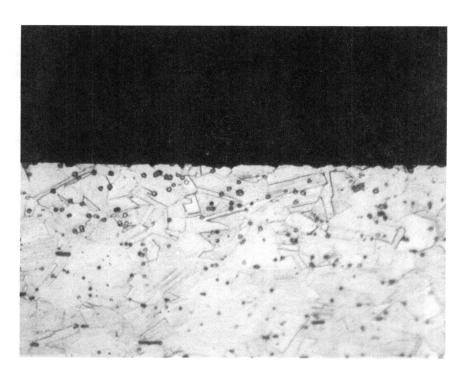


Fig. 19. Moist MCV resin exposure - stainless steel (301) sectioned metallograph at 400x.

## 6.0 <u>DISCUSSION</u> AND CONCLUSIONS

There were several factors present in this study which prevented a thorough interpretation of the results.

- 1. Original specimen weights, dimensions, and surface finishes were not available.
- 2. Reference specimens were available for the 321 and 347 specimens, but not for 316L or the specimen exposed to moist MCV resin. In addition, the exact material or test history was not well documented for the stainless steel strip specimen exposed to moist MCV resin.
- 3. The amount of water and MCV resin prepared was not held constant when preparing the vials. This prevented a quantitative comparison of the results obtained from the blank vial to the test vials.
- 4. Too many unknown factors were present in the blank vial which may have affected the results. A second blank without MCV resin would have been required to differentiate between the effects of the glass and plastic cap from that of the MCV resin. This would have helped to identify the origin of the various cations and metals found in the blank.
- 5. The MCV resin did not maintain a steady iodine concentration in the water solution. Because of this, the iodine concentrations gradually increased to 236 mg/l in the blank vial and over 69 mg/l in the test vials. This was unexpected and resulted in an iodine environment that differs somewhat from what is expected in the IWS (2-6 mg/l l<sub>2</sub>).

Despite the numerous faults in the study, the investigation offered a good insight into long-term effects of iodine exposure. Aqueous and vapor iodine exposure is a concern for the IWS which is expected to meet a 30-year life. In addition, the materials in this study were of interest to this system. The current baseline material for the IWS is 316L for the lines and bellows tank. In general, the opportunity to examine specimens exposed to an iodine environment for this length of time was unique and provided a chance to evaluate long-term iodine exposure even if the test conditions were not exactly representative of the actual system.

From the results of this study, it is concluded that aqueous exposure to iodine did not significantly effect the stainless steels tested. The quantity of metals released by the specimens exposed to the aqueous environment resulted in a corrosion rate on the order of 1 x10<sup>-5</sup> mpy. This rate would have a negligible effect on the structural integrity of the IWS. The specimen exposed to moist MCV resin was determined to be SS 301. Exposure of this alloy to moist MCV resin and air did result in noticeable intergranular corrosion; however, it only penetrated the first grain boundary layer.

# 7.0 REFERENCES

- American Public Health Association, American Water Works Association, Water Pollution Control Federation: Standard Methods for the Examination of Water & Wastewater, 17th Edition, American Public Health Association.
- 2) EPA Methods, Chemical Analysis of Water and Wastes, 1979.

Ť.			
-			
:			
-			

REPORT I	DOCUMENTATION PA	AGE	Form Approved OMB No. 0704-0188
maintaining the data needed, and completing and	reviewing the collection of information. Send of Vashington Headquarters Services, Directorate for	comments regarding this burden estimate or Information Operations and Reports, 1215, Washington, DC 20503.	ions, searching existing data sources, gathering and r any other aspect of this collection of information, Jefferson Davis Highway, Suite 1204, Arlington, VA
AGENCY USE ONLY (Leave blank)	2. REPORT DATE  January 1992	3. REPORT TYPE AND DATES Technical Memora	covered andum
4. TITLE AND SUBTITLE Long-Term Corrosion E Shuttle Iodinated Resin	Evaluation of Stainless Sto and Water	eels in Space	5. FUNDING NUMBERS
6. AUTHOR(S) Douglas D. Krohn			
7. PERFORMING ORGANIZATION NAI Propulsion and Power National Aeronautics ar Johnson Space Center Houston, Texas 7705	nd Space Administration	<b>1</b>	PERFORMING ORGANIZATION REPORT NUMBER S-660
9. SPONSORING/MONITORING AGEI National Aeronautics a Washington, D. C. 209	nd Space Administration		SPONSORING/MONITORING AGENCY REPORT NUMBER NASA-TM-104743
11. SUPPLEMENTARY NOTES			
12a. DISTRIBUTION / AVAILABILITY STA Unclassified/Unlimited Subject Category 26	Publicly Available		b. DISTRIBUTION CODE
Water System (IWS) for effects of general and lo candidate materials. In specimens in a solution 1990, the solution was confeach specimen was estimated the extent of general and steels was negligible and 316L performed the best environment. In addition (unspecified alloy) was estimated the steel that this alloy was stainless to the first grain boundar iodine, metallography,	of deionized water and the hemically analyzed to deter examined with scanning elective process. This exist is the specimens exposed to moist microbial discontact of the metal to the alloy was not known, energy layer.	the IWS has a life require he a long period have not center immersed stainles Space Shuttle microbial rmine the level of corros ctron microscopy and mamination showed that the first grain boundary lay als proved to be compated to aqueous iodine, a standard check valve resin and a e resin as well as to the gy dispersive spectrosconular corrosion found or	ement of 30 years, but the been determined for the ss steel 316L, 321, and 347 check valve resin. In April ion formed, and the surface retallography to determine the attack on the stainless er. Of the three alloys, ible with an aqueous iodine ainless steel specimen ir for a comparable period.
scanning electron micro	oscopy, stainless steel	,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	16. PRICE CODE
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT UL

<del>.</del>		
<b>\</b>		
<del>.</del>		
-		

Langley Research Center Hampton, VA 23665 185/Technical Library (3)

